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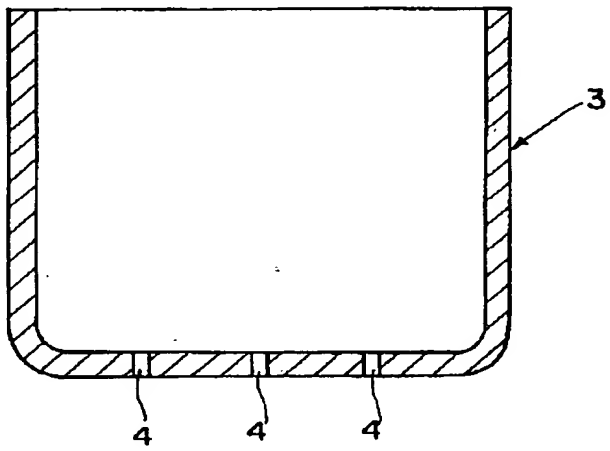
(54)【発明の名称】シリコン単結晶引上装置

(57)【要約】

【課題】　シリコン単結晶引上装置において、黒鉛ルツボに石英ルツボを装填する際に、石英ルツボの外側と黒鉛ルツボ内側との間隙に存在するガスが圧縮され、該両ルツボの上部隙間から吹き出し、この時黒鉛粉末が吹き出すガスに同伴して舞い上がり周囲を汚染するのを防止する。

【解決手段】　シリコン融液を収容する石英ルツボと該石英ルツボを保持する黒鉛ルツボとを少なくとも備え、石英ルツボ中のシリコン融液にシリコン種結晶を浸し、所望の単結晶を引上げるシリコンウエハ単結晶引上装置において、前記黒鉛ルツボに石英ルツボを装填する際に、両ルツボ間の空隙領域に存在するガスを逃がすための貫通開口が黒鉛ルツボの底部に少なくとも１個設けられている。



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【特許請求の範囲】

【請求項１】　シリコン融液を収容する石英ルツボと該石英ルツボを保持する黒鉛ルツボとを少なくとも備え、石英ルツボ中のシリコン融液にシリコン種結晶を浸し、所望の単結晶を引上げるシリコンウエハ単結晶引上装置において、

前記黒鉛ルツボ内に石英ルツボを装填する際、両ルツボ間の空隙領域に存在するガスを逃がすための貫通開口が、前記黒鉛ルツボの底部に少なくとも１個設けられていることを特徴とするシリコンウエハ単結晶引上装置。

【請求項２】　前記黒鉛ルツボ底部に設けられる貫通開口の数が１乃至１０個、その直径が５ｍｍ以下であることを特徴とする請求項１に記載されたシリコンウエハ単結晶引上装置。

【請求項３】　前記貫通開口の数が１乃至１０個、その直径が２乃至５ｍｍであることを特徴とする請求項２に記載されたシリコンウエハ単結晶引上装置。

【請求項４】　前記貫通開口が、黒鉛ルツボのルツボ底中心部近傍に配置されていることを特徴とする請求項１乃至請求項３のいずれかに記載されたシリコンウエハ単結晶引上装置。

【請求項５】　前記黒鉛ルツボの内部側壁に、その底部から上部に向けて縦状細溝が設けらていることを特徴とする請求項１乃至請求項４のいずれかに記載されたシリコンウエハ単結晶引上装置。

【請求項６】　前記黒鉛ルツボの口径が１２インチ以上であることを特徴とする請求項１乃至請求項５のいずれかに記載されたシリコンウエハ単結晶引上装置。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明はシリコンウエハ単結晶引上装置に関し、より詳細には大口径大容積のシリコン単結晶引上に好適に使用される大型ルツボを備えたシリコンウエハ単結晶引上装置に関する。

【０００２】

【従来の技術】従来、半導体単結晶、例えば高純度シリコン単結晶を製造する工業的方法としては、黒鉛製のルツボによって保持された石英製ルツボに多結晶シリコンを投入し、該多結晶シリコンをヒータによって加熱融解し、このシリコン融液に浸した種結晶を徐々に引き上げつつ凝固させて、単結晶インゴットを製造するいわゆるチョクラルスキー法が一般に採用されている。

【０００３】この製造方法では、例えば、図４に示したようなシリコンウエハ単結晶引上装置が用いられいる。即ち、シリコンウエハ単結晶引上装置１は、黒鉛製のルツボ３によって保持された石英製ルツボ２と、石英製ルツボ２投入された多結晶シリコンを加熱融解するヒータ６と、前記ヒータ６の外側を覆う断熱材７と、前記ヒータ６によって溶融したシリコン融液に浸した種結晶を徐々に引き上げる引上げる引上げ手段９と、装置内にアルゴンガス等の不活性ガス１１を供給する供給手段（図示せず）と、前記黒鉛製のルツボ３及び石英製ルツボ２を回転させる回転手段１２とから構成されている。

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【０００４】そして、シリコンウエハ単結晶引上装置１の石英製ルツボ２に高純度の多結晶シリコンを入れ、更にこの石英ルツボ２を保持用黒鉛ルツボ３に装填し、黒鉛発熱体のヒータ６で加熱融解する。このとき、シリコンの融点は１４２０℃と石英の軟化点に近いが、前記石英ルツボ２は黒鉛ルツボ３で保持されているため、石英ルツボ２が変形等を起こすことはない。この多結晶シリコンの加熱融解は、通常、アルゴンガス等の不活性ガス１１の雰囲気中で行われ、融解後融液表面に種子結晶１０を接触させ結晶を作成する。なお、図４中、８はシリコンの融液を示している。この単結晶引上げに際し、種子結晶部１０からの転位を除去するため、通常直径５ｍｍ以下の部分を形成（ネッキング）した後、直径を増大し所定の大きさにする。そして一定径で結晶を作成した後、徐々に直径を小さくして、融液から切り離し、インゴットとしている。

【０００５】

【発明が解決しようとする課題】ところが、多結晶シリコンを溶融して単結晶インゴットを製造するため、上記のように石英製ルツボに高純度の多結晶シリコンを入れ、この石英ルツボを保持用黒鉛ルツボに装填する際に、石英ルツボの外側と黒鉛ルツボ内側との間隙が小さいために、石英ルツボと黒鉛ルツボの間に存在するガスが装填時に圧縮され、該両ルツボ間の僅かな上部隙間から吹き出す。このとき、黒鉛ルツボの内壁面に付着していた黒鉛粉末が吹き出すガスに同伴して舞い上がり、上部隙間から装置内部に吹き出すため、装置内部を汚染するという不都合がしばしば生じていた。この不都合は、必要とされるウエハが小口径の場合には、小型のインゴットで済むため、石英ルツボや黒鉛ルツボも小型であり、その黒鉛粉末の舞い上がりはそれほど大きな問題ではなかった。しかし、今日ウエハの大口径化に伴い、石英ルツボ及び黒鉛保持用ルツボが大型化するにつれてその影響が顕在化し、深刻な問題となっている。

【０００６】本発明は上記課題を解決するためになされた発明であって、石英ガラスの装填が容易であるとと共に、炭素粉末による汚染を防止することができる改良された黒鉛ルツボを提供することを目的とするものである。

【０００７】

【課題を解決するための手段】本発明によれば、シリコン融液を収容する石英ルツボと該石英ルツボを保持する黒鉛ルツボとを少なくとも備え、石英ルツボ中のシリコン融液にシリコン種結晶を浸し、所望の単結晶を引上げるシリコンウエハ単結晶引上装置において、前記黒鉛ルツボ内に石英ルツボを装填する際、両ルツボ間の空隙領域に存在するガスを逃がすための貫通開口が、前記黒鉛

ルツボの底部に少なくとも１個設けられていることを特徴とするシリコンウエハ単結晶引上装置が提供される。

【０００８】本発明は、石英ルツボと石英ルツボ保持用黒鉛ルツボとを備えた、いわゆるチョクラルスキー法によるシリコンウエハ単結晶引上装置において、石英ルツボ装填の際に、両ルツボ間の空隙領域に存在するガスを下方に逃がすための貫通開口を黒鉛ルツボの底部に１個以上設ける点が構成上の特徴である。既に述べたように、従来の装置では、石英ルツボと黒鉛ルツボの間隙に存在するガスを逃がす逃げ場は両ルツボの間隙部しかないため、ガスが石英ルツボ装填時に圧縮され、該圧縮されたガスは両ルツボの上部隙間から吹き出し、この時黒鉛粉末が吹き出すガスに同伴して舞い上がり周囲を汚染するという不都合が生じていた。

【０００９】これを、黒鉛粉を含んだガスを黒鉛ルツボの底部から下方の向けて逃がすことにより、ルツボ上部から上方にあるいは側面から外側方向に逃がす場合に比べて、石英ルツボ内の溶融シリコン汚染の程度を著しくする低減することができる。特に、大型石英ルツボ及び黒鉛保持用ルツボの場合、その効果が顕著に現れる。即ち、黒鉛粉を含んだガスは黒鉛ルツボの底部から下方の向けて逃がされるため、黒鉛粉は装置内部下方に浮遊することになる。ガス中に浮遊する黒鉛粉等粉塵の浮遊量はその高さに依存して指数関数的に減少するため、高さの高い大型ルツボの場合、上方あるいは側方から逃がす場合に比べて、ルツボの上部に浮遊する黒鉛粉は著しく減少し、ルツボ内部に侵入する黒鉛粉は著しく減少する。

【００１０】本発明は、黒鉛ルツボの底部にガス抜き用の孔を設けるという構成上簡単な発明であるが、従来の公知黒鉛ルツボには、ヒーターからの輻射熱を活用するためその上部側壁に開口を設けるもの（特開平４－１２０８５号公報）、黒鉛ルツボの湾曲部内面側に蓄積した金属類がシリコン融液及び凝固直後のシリコン単結晶に混用するのを防止するため、その側壁に開口を設けるもの（特開平５－２０８８９０号公報）等はあっても、底部にガス抜き用開口を設けた黒鉛ルツボは、本発明者の知る限り本発明出願以前には知られていない。また上記本発明のルツボに、更に、例えば図３に示すようにルツボ内側側壁に底部から上部に向けて縦に細溝を設けたものは石英ルツボ装填時のガス抜きがよりスムーズとなり好適である。更に、前記石英ルツボの黒鉛ルツボへの装填を減圧下に行えばより一層効果的に無汚染装填を実施することができる。

【００１１】

【発明の実施の形態】本発明のシリコンウエハ単結晶引上装置に用いられる黒鉛ルツボとしては、該ルツボ底部に石英ルツボ装填時のガス抜き孔が設けられている以外は、公知の黒鉛ルツボと同様のサイズ、形状、構造のものを用いることが出来る。ルツボサイズとしては、直径４インチ以上の単結晶インゴットの引上げに使用される口径１２インチ以上、より好ましくは１２インチ以上のインゴット引上げに使用される口径２４インチ以上のものが本発明の目的とする効果が顕著に現れるため好適である。

【００１２】本発明のシリコンウエハ単結晶引上装置に用いられる黒鉛ルツボ及び石英ルツボを図１及び図２に基づいて詳細に説明する。本発明の黒鉛ルツボ３の必須構成要件であるルツボ底部に設けられる貫通開口４は、図１に示すように、石英ルツボ２装填時にガスがスムーズに排出できるように、その数及び口径が設定され、黒鉛ルツボ３のサイズに依存して適宜設定されるが、通常、直径５ｍｍ以下、好ましくは直径２乃至５ｍｍの貫通孔を１０個以下、より好ましくは１乃至５個設ける。黒鉛ルツボ３の機械的強度をある程度以上に維持し、繰り返し使用可能な耐久力を具備させるためには、貫通開口４の孔径はなるべく小く、またその個数はなるべく少なく、石英ルツボ２の装填時にガスが黒鉛ルツボ３の底部からスムーズに排出できる必要最低限度に設定することが好ましい。

【００１３】ルツボ底部に設けられる貫通開口４が複数の場合、その配置はランダムに配置されても良く、底部中心から同心円状に配置されても良い。本発明においては、貫通開口４の配置位置は、単純圧縮応力のみが働き他の方向の応力成分が作用しないルツボ底中心部近傍に配置することが、その機械的強度を保持する上から好ましい。

【００１４】本発明の黒鉛ルツボ３には上記ルツボ底部の貫通開口４に加えて更に、例えば図３に示すようにルツボ内側側壁に底部から上部に向けて縦に細溝５を設けるても良く、このよう黒鉛ルツボ３に貫通開口と縦状溝５とを形成したものは石英ルツボ装填時のガス抜きがよりスムーズとなり好適である。黒鉛ルツボ３の側面に設けられる該縦溝５の本数、サイズ、形状等は、黒鉛ルツボ３の機械的強度を大きく低下させ無い限度において任意に設定できるが、縦溝５の配置としては、ルツボ中心軸に対象に複数本、例えば２乃至４本設けることが好ましく、溝の深さは２ｍｍ乃至４ｍｍ程度、形状は半円形乃至半楕円形が好ましい。

【００１５】本発明のシリコンウエハ単結晶引上装置の稼働に際し、多結晶シリコンが収容された石英ルツボ２の黒鉛ルツボへ３の装填は、それ自体公知のルツボ装填装置を用いて、通常アルゴン等の不活性ガス雰囲気下に、常圧で行われるが、この石英ルツボ２装填を、例えば、減圧下での操作が可能なルツボ装填装置を用いて減圧下に行えばより一層効果的に無汚染装填を実施することができる。

【００１６】

【実施例】「実施例１、比較例１」３００ｍｍφシリコン単結晶引上用装置において、３０インチφ石英ルツボ

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（外径７６０mm、重量約４０ｋｇ）及び底部に２mmφの貫通孔を４個、図２に示すように配設した石英ルツボ保持用黒鉛ルツボ（内径７６１mm、重量約１００ｋｇ：実施例１）と、ルツボ底部に貫通孔が無い以外は本発明の黒鉛ルツボ（実施例１）と全く同サイズ同型の黒鉛ルツボ（比較例１）を夫々用意した。

【００１７】先ず引上げチャンバー内に保温部材、ヒータ及び上記本発明の黒鉛ルツボ（実施例１）をセットし、次いで石英ルツボを黒鉛ルツボ（実施例１）に挿入セットし、この時の装填容易性及び黒鉛粉の飛散状態を観察評価した（実施例１）。次に、本発明の黒鉛ルツボに替えて底部に貫通開口を有さない従来の黒鉛ルツボ（比較例１）をセットし、次いで前記と同様に石英ルツボを挿入セットし、この時の装填容易性及び黒鉛粉の飛散状態を同様に観察評価した（比較例１）。

【００１８】従来の黒鉛ルツボ（比較例１）では、石英ルツボを挿入する際、黒鉛ルツボ内のガスが圧縮され、この圧縮ガスの弾性反発力により該黒鉛ルツボに石英ルツボをスムーズに装填することが妨げられ、またこのとき、両ルツボの上部隙間から黒鉛粉がガスと共に吹き出し、これが周囲に飛散して周囲を汚染した。これに対し、本発明の黒鉛ルツボの場合（実施例１）は、底部の細孔からガスがスムーズに抜けるため石英ルツボがスムーズに沈み、挿入セットが容易にできただけでなく、ガスの上部からの吹き出しも殆どなく、従って周囲の汚染も殆ど認められなかった。

【００１９】次に、石英ルツボ内の汚染程度を比較評価するため、実施例１、比較例１共に石英ルツボの内部底部に清浄な５インチφシリコンウエハを置き、石英ルツボ装填後に夫々ルツボ内のウエハを回収し、夫々ウエハに付着したパーティクルの数を測定し、汚染の程度を比較評価した。その結果を表１に示す。また夫々のウエハに付着したパーティクルを回収して分析したところ、比較例１でのウエハ付着パーティクルには５０％以上の割合で炭素が検出された。これに対し、実施例１の場合は殆ど炭素は検出されなかった。その結果を表１に示す。なお、測定したパーテイクルは、径が０．２μm以上のものを測定対象とし、石英ルツボセット前後のパーテイクル数の増加した個数を数えた。

【００２０】

【表１】

	付着パーテイクル数（ΔN）	パーテイクル分析結果
実施例１	10個未満／ウエハ	炭素不検出（検出限界以下）
比較例１	10個以上／ウエハ	炭素検出

【００２１】「実施例２，比較例２」実施例１で用いた本発明の黒鉛ルツボと比較例１の従来の黒鉛ルツボを用い、夫々、下記条件で溶融シリコンを入れた石英ルツボから３００mmφシリコン単結晶を引き上げた（実施例２，比較例２）。このときの引上条件は、以下の通りである。

チャージ量（石英ルツボへのシリコン装填量）：　約１８０ｋｇ
引上結晶：　＜１００＞
引上速度：　０．３～０．８mm／ｍｉｎ
結晶回転（石英ルツボの回転数）：　８～１５ｒｐｍ
炉内圧：　Ａｒガス雰囲気３０～１００Ｔｏｒｒ

夫々の黒鉛ルツボについて各１０回引上を実施し、得られたインゴットの不良品率を検査した。その結果を表２に示す。

【００２２】

【表２】

	良品率（%）	備考欄
実施例２	80以上	異物付着による不良品殆ど無し
比較例２	40～60	異物付着で引上初期に不良品頻度大

【００２３】従来品ルツボ（比較例２）で引上た場合は、引上げ途中において異物の付着による有転位化現象が顕著に見られた。この付着物を採取し分析したところ殆どの場合炭素が検出された（異物はＳｉＣと推定される。）

これに対し、本発明品の場合（実施例２）は殆ど異物の付着が見られず、不良品が発生した場合も殆どが他の原因によるものであった。

【００２４】また引き上げたシリコン結晶インゴットの尾部からウエハを切り出し（サンプルａ、ｂ、ｃ、Ａ、Ｂ、Ｃとして、それぞれのインゴットから３つ切り出し）、赤外線吸収スペクトル測定法によって炭素濃度を

7　　8

測定した。その結果、従来品ルツボの場合は、１０'' ａｔｏｍｓ／ｃｍ' レベルの炭素が検出された。これに対し、本発明品の場合は、１０'' ａｔｏｍｓ／ｃｍ' 以下であった。その結果を表３に示す。

【００２５】

【表３】

	サンプル	炭素濃度
実施例２	a	$< 5 \times 10^{15}$ atoms／cm^3
	b	$< 5 \times 10^{15}$ atoms／cm^3
	c	8.1×10^{15} atoms／cm^3
比較例２	A	22×10^{17} atoms／cm^3
	B	6.5×10^{16} atoms／cm^3
	C	1.2×10^{17} atoms／cm^3

【００２６】

【発明の効果】本発明のシリコンウエハ単結晶引上装置は、その黒鉛ルツボが上述した通りの特定構成を有するものであるため、石英ルツボの黒鉛ルツボへの装填が容易であるという効果を奏するものである。また石英ルツボ内容物の炭素粉末による汚染が回避され、従来の黒鉛ルツボを使用した装置に比較して得られるシリコン単結晶インゴットの不良品率が顕著に改善される等の利点を有し、特に直径３００mm以上の大口径シリコン単結晶引上げに有用である。

【図面の簡単な説明】

【図１】図１は、本発明のシリコン単結晶引上装置で用いられる支持用黒鉛ルツボの断面略図である。

【図２】図２は、従来のシリコン単結晶引上装置で用いられる支持用黒鉛ルツボの断面略図である。

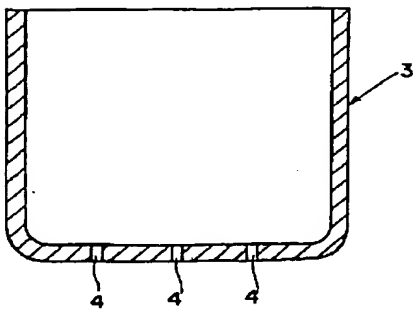
【図３】図３は、本発明のシリコン単結晶引上装置で用いられる、更にガス抜き用縦状溝が設けられた黒鉛ルツボの部分断面略図である。

【図４】チョクラルスキー法で用いられる単結晶引上装置を説明するための概略図である。

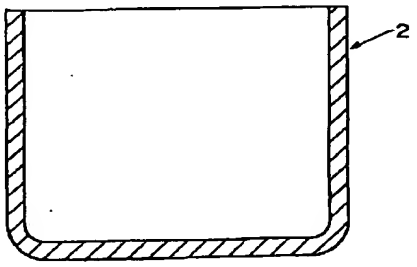
【符号の説明】

1　単結晶引上装置本体
2　石英ルツボ
3　黒鉛ルツボ
4　貫通開口
5　細溝
6　ヒータ
7　断熱材
8　シリコン融液
9　引上げ手段
１０　種結晶
１１　不活性ガス
１２　回転手段

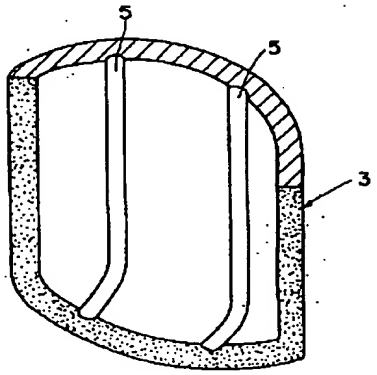
【図１】



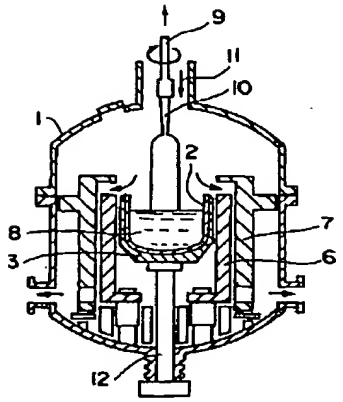
【図２】



【図３】



【図４】



* NOTICES *

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Epitome

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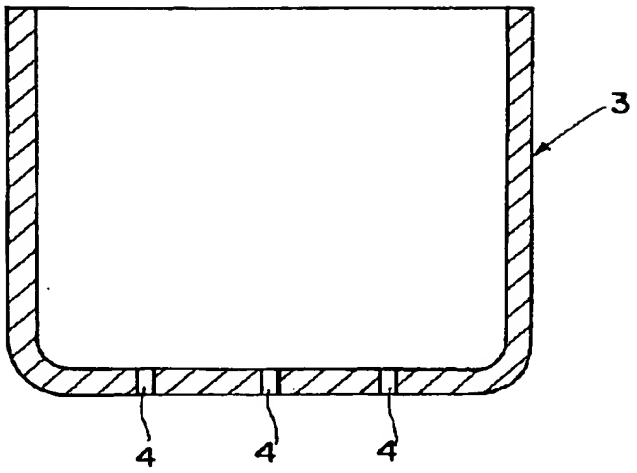
(57) [Abstract]
[Technical problem] In silicon single crystal raising equipment, in case a graphite crucible is loaded with a quartz crucible, it prevents the gas which exists in the gap of the outside of a quartz crucible and the graphite crucible inside being compressed, and accompanying to the blowdown and the gas by which graphite powder blows off at this time, soaring from the up clearance between these both crucibles, and polluting a perimeter.
[Means for Solution] It has at least the quartz crucible which holds silicon melt, and a graphite crucible holding this quartz crucible, and silicon seed crystal is dipped in the silicon melt in a quartz crucible, and in the silicon wafer crystal-pulling equipment which pulls up a desired single crystal, in case said graphite crucible is loaded with a quartz crucible, penetration opening for missing the gas which exists in the opening field between both crucibles is prepared in at least one pars basilaris ossis occipitalis of a graphite crucible.

[Translation done.]



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CLAIMS

[Claim(s)]
[Claim 1] Silicon-wafer crystal-pulling equipment with which penetration opening for missing the gas which exists in the opening field between both crucibles is characterized by to be prepared in at least one pars basilaris ossis occipitalis of said graphite crucible in the silicon-wafer crystal-pulling equipment which is equipped with the quartz crucible which holds silicon melt, and the graphite crucible holding this quartz crucible at least, dips silicon seed crystal in the silicon melt in a quartz crucible, and pulls up a desired single crystal in case it loads with a quartz crucible into said graphite crucible.
[Claim 2] Silicon wafer crystal-pulling equipment with which the number of penetration openings prepared in said graphite crucible pars basilaris ossis occipitalis was indicated by claim 1 characterized by 1 thru/or ten

pieces, and the diameter of those being 5mm or less.
[Claim 3] Silicon wafer crystal-pulling equipment with which the number of said penetration openings was indicated by claim 2 characterized by 1 thru/or ten pieces, and the diameter of those being 2 thru/or 5mm.
[Claim 4] Silicon wafer crystal-pulling equipment indicated by either claim 1 to which said penetration opening is characterized by being arranged near the crucible bottom core of a graphite crucible thru/or claim 3.
[Claim 5] the internal side attachment wall of said graphite crucible -- the upper part from the pars basilaris ossis occipitalis -- turning -- a length-like striation -- preparing -- ******** -- the silicon wafer crystal-pulling equipment indicated by either claim 1 characterized by things thru/or claim 4.
[Claim 6] Silicon wafer crystal-pulling equipment indicated by either claim 1 characterized by the aperture of said graphite crucible being 12 inches or more thru/or claim 5.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the silicon wafer crystal-pulling equipment which equipped the detail with the large-sized crucible used suitable for silicon single crystal raising of the diameter size volume of macrostomia more about silicon wafer crystal-pulling equipment.
[0002]
[Description of the Prior Art] As the industrial approach of

manufacturing a semi-conductor single crystal, for example, a high-purity-silicon single crystal, polycrystalline silicon is conventionally thrown into the crucible made from a quartz held by the crucible made from a graphite, heating fusion of this polycrystalline silicon is carried out at a heater, it is made to solidify, pulling up gradually the seed crystal dipped in this silicon melt, and, generally the so-called Czochrlski method which manufactures a single crystal ingot is adopted.

[0003] By this manufacture approach, for example, silicon wafer crystal-pulling equipment as shown in drawing 4 is used, and it is. Namely, the crucible 2 made from a quartz by which silicon wafer crystal-pulling equipment 1 was held by the crucible 3 made from a graphite, Crucible 2 made from a quartz The heater 6 which carries out heating fusion of the thrown-in polycrystalline silicon, A pull-up means 9 to pull up to pull up gradually the seed crystal which dipped in the silicon melt which fused the outside of said heater 6 at the wrap heat insulator 7 and said heater 6, It consists of a supply means (not shown) to supply inert gas 11, such as argon gas, in equipment, and a revolution means 12 to rotate the crucible 3 and the crucible 2 made from a quartz made from said graphite.

[0004] And the polycrystalline silicon of a high grade is put into the crucible 2 made from a quartz of silicon wafer crystal-pulling equipment 1, the graphite crucible 3 for maintenance is further loaded with this quartz crucible 2, and heating fusion is carried out at the heater 6 of a graphite heating element. Although the melting point of silicon is close to the softening temperature of 1420 degrees C and a quartz at this time, since said quartz crucible 2 is held by the graphite crucible 3, the quartz crucible 2 does not cause deformation etc. Heating fusion of this polycrystalline silicon is usually performed in the ambient atmosphere of inert gas 11, such as argon gas, a seed crystal 10 is contacted on the melt front face after fusion, and a crystal is created. In addition, eight show the melt of silicon among drawing 4 . In order to remove the rearrangement from the seed crystal section 10 on the occasion of this crystal pulling, it increases and a diameter is made into predetermined magnitude, after usually forming a part with a diameter of 5mm or less (necking). And after creating a crystal with the diameter of fixed, a diameter is gradually made small, and it separates from melt and is considering as the ingot.

[0005]

[Problem(s) to be Solved by the Invention] However, in order to fuse polycrystalline silicon and to manufacture a single crystal ingot, in case

the polycrystalline silicon of a high grade is put into the crucible made from a quartz as mentioned above and the graphite crucible for maintenance is loaded with this quartz crucible, since the gap of the outside of a quartz crucible and the graphite crucible inside is small, the gas which exists between a quartz crucible and a graphite crucible is compressed at the time of loading, and blows off from few up clearances between these both crucibles. Since it accompanied to the gas by which the graphite powder adhering to the internal surface of a graphite crucible blows off at this time, it soared and it blew off from an up clearance inside equipment, the inconvenience of polluting the interior of equipment often arose. since this inconvenience can be managed with a small ingot when the wafer needed is a diameter of a header -- a quartz crucible and a graphite crucible -- small -- that graphite powder -- soaring -- it was not so big a problem. However, with diameter[of macrostomia]-izing of a wafer, the effect actualizes as a quartz crucible and the crucible for graphite maintenance are enlarged, and it has been a serious problem today.

[0006] It is invention made in order that this invention might solve the above-mentioned technical problem, and aims at both offering the improved graphite crucible which can prevent contamination according to carbon powder as if loading of quartz glass is easy.

[0007]

[Means for Solving the Problem] According to this invention, it has at least the quartz crucible which holds silicon melt, and a graphite crucible holding this quartz crucible. In the silicon wafer crystal-pulling equipment which dips silicon seed crystal in the silicon melt in a quartz crucible, and pulls up a desired single crystal In case it loads with a quartz crucible into said graphite crucible, the silicon wafer crystal-pulling equipment with which penetration opening for missing the gas which exists in the opening field between both crucibles is characterized by being prepared in at least one pars basilaris ossis occipitalis of said graphite crucible is offered.

[0008] This invention is the description on a configuration of the point of preparing penetration opening for missing caudad the gas which exists in the opening field between both crucibles in the case of quartz crucible loading in one or more partes basilaris ossis occipitalis of a graphite crucible, in the silicon wafer crystal-pulling equipment equipped with the quartz crucible and the graphite crucible for quartz crucible maintenance by the so-called Czochrlski method. Since the refuge which misses the gas which exists in the gap of a quartz crucible and a graphite crucible

with conventional equipment had only the gap section of both crucibles as already stated, gas was compressed at the time of quartz crucible loading, it accompanied to the blowdown and the gas by which graphite powder blows off at this time, the this compressed gas soared from the up clearance between both crucibles, and the inconvenience of polluting a perimeter had produced it.

[0009] a lower part turning the gas which contained graphite powder for this, and missing it from the pars basilaris ossis occipitalis of a graphite crucible, -- the upper part from the crucible upper part -- or compared with the case where it misses in the direction of an outside, extent of the melting silicon contamination in a quartz crucible is made remarkable from a side face -- it can decrease. Especially, in the case of a large-sized quartz crucible and the crucible for graphite maintenance, the effectiveness shows up notably. That is, since a lower part turns the gas containing graphite powder and it is missed from the pars basilaris ossis occipitalis of a graphite crucible, graphite powder will float in the interior lower part of equipment. In order that the amount of floating of dust, such as graphite powder which floats in gas, may decrease exponentially depending on the height, in the case of a large-sized crucible with high height, compared with the case where it misses from the upper part or the side, the graphite powder which floats in the upper part of a crucible decreases in number remarkably, and the graphite powder which trespasses upon the interior of a crucible decreases in number remarkably.

[0010] Although this invention is easy invention on the configuration of preparing the hole for deflation in the pars basilaris ossis occipitalis of a graphite crucible What prepares opening in the up side attachment wall in order to utilize the radiant heat from a heater for the conventional well-known graphite crucible (JP,4-12085,A), In order to prevent that the metals accumulated in the bend inner surface side of a graphite crucible use together to the silicon single crystal immediately after silicon melt and coagulation, Even if there are some (JP,5-208890,A) which prepare opening in the side attachment wall, the graphite crucible which prepared opening for deflation in the pars basilaris ossis occipitalis is not known before this invention application, as far as this invention person gets to know. Moreover, what prepared the striation in the crucible of above-mentioned this invention perpendicularly towards the upper part further at the crucible inside side attachment wall from the pars basilaris ossis occipitalis as shown in drawing 3 becomes [the deflation at the time of quartz crucible loading] more smooth and is

suitable. Furthermore, if loading to the graphite crucible of said quartz crucible is performed to the bottom of reduced pressure, non-polluted loading can be carried out much more effectively.

[0011]

[Embodiment of the Invention] As a graphite crucible used for the silicon wafer crystal-pulling equipment of this invention, except that the deflation hole at the time of quartz crucible loading is prepared in this crucible pars basilaris ossis occipitalis, the thing of the same size as a well-known graphite crucible, a configuration, and structure can be used. As crucible size, the aperture of 12 inches or more used for a pull-up of a single crystal ingot with a diameter of 4 inches or more, since the effectiveness which a thing with an aperture of 24 inches or more more preferably used for an ingot pull-up of 12 inches or more makes the object of this invention shows up notably, it is suitable.

[0012] The graphite crucible and quartz crucible which are used for the silicon wafer crystal-pulling equipment of this invention are explained to a detail based on drawing 1 and drawing 2 . although the number and aperture are set up and it is suitably set up depending on the size of a graphite crucible 3 so that gas can discharge smoothly at the time of quartz crucible 2 loading as the penetration opening 4 prepared in the crucible pars basilaris ossis occipitalis which are the requirements for an indispensable configuration of the graphite crucible 3 of this invention is shown in drawing 1 -- usually -- the diameter of 5mm or less -- desirable -- a diameter 2 thru/or a 5mm breakthrough -- ten or less pieces -- more -- desirable -- 1 -- or five pieces are prepared. The mechanical strength of a graphite crucible 3 is maintained to some extent above, in order to make an usable strength provide repeatedly, if possible, the aperture of the penetration opening 4 has if possible little small ** and its number, and it is desirable to set it as the need minimum which gas can discharge smoothly from the pars basilaris ossis occipitalis of a graphite crucible 3 at the time of loading of the quartz crucible 2.

[0013] When the penetration opening 4 prepared in a crucible pars basilaris ossis occipitalis is plurality, the arrangement may be arranged at random and may be arranged in the shape of a concentric circle from a pars-basilaris-ossis-occipitalis core. As for the arrangement location of the penetration opening 4, in this invention, it is [from / when holding the mechanical strength] desirable to arrange near the crucible bottom core where only simple compression stress works and the stress component of other directions does not act.

[0014] It becomes [in addition to the penetration opening 4 of the above-mentioned crucible pars basilaris ossis occipitalis in the graphite crucible 3 of this invention, / ** which forms a striation 5 perpendicularly towards the upper part on a crucible inside side attachment wall from a pars basilaris ossis occipitalis as shown further at drawing 3 is also good, and / what formed penetration opening and ****** 5 in the such graphite crucible 3] more smooth [the deflation at the time of quartz crucible loading] and is suitable. Although the number of this fluting 5 prepared in the side face of a graphite crucible 3, size, a configuration, etc. can be set as arbitration in the limit which reduces the mechanical strength of a graphite crucible 3 greatly, and is not, as arrangement of a fluting 5, it is desirable to a crucible medial axis two or more, 2 [for example,], thru/or to prepare four in an object, and a semicircle thru/or the half-ellipse form of a tooth depth are [2mm thru/or about 4mm, and a configuration] desirable.

[0015] Although usually carried out by ordinary pressure to the bottom of inert gas ambient atmospheres, such as an argon, to the graphite crucible of the quartz crucible 2 in which polycrystalline silicon was held using a crucible gun charger with loading of 3 well-known in itself on the occasion of operation of the silicon wafer crystal-pulling equipment of this invention, if this quartz crucible 2 loading is performed to the bottom of reduced pressure using the crucible gun charger in which the actuation for example, under reduced pressure is possible, non-polluted loading can be carried out much more effectively.

[0016]

[Example] In the equipment for an example 1 and "example 1 of comparison" 300mmphi silicon single crystal raising The graphite crucible for quartz crucible maintenance which arranged the breakthrough of 2mmphi in the 30 inch phi quartz crucible (the outer diameter of 760mm, weight of about 40kg), and the pars basilaris ossis occipitalis as shown in four pieces and drawing 2 (the bore of 761mm, weight of about 100kg: example 1), Except that there was no breakthrough in a crucible pars basilaris ossis occipitalis, the graphite crucible (example 1 of a comparison) of same size isomorphism was completely prepared with the graphite crucible (example 1) of this invention, respectively.

[0017] The graphite crucible (example 1) of an incubation member, a heater, and above-mentioned this invention was first set in the pull-up chamber, subsequently to a graphite crucible (example 1) the insertion set of the quartz crucible was carried out, and observation assessment

of the loading ease at this time and the scattering condition of graphite powder was carried out (example 1). Next, the conventional graphite crucible (example 1 of a comparison) which changes to the graphite crucible of this invention and does not have penetration opening at the pars basilaris ossis occipitalis was set, subsequently the insertion set of the quartz crucible was carried out like the above, and observation assessment of the loading ease at this time and the scattering condition of graphite powder was carried out similarly (example 1 of a comparison).

[0018] When inserting a quartz crucible, the gas in a graphite crucible was compressed, loading this graphite crucible with a quartz crucible smoothly according to the elastic repulsive force of this compressed gas was barred, and at this time, graphite powder dispersed in the blowdown with gas from the up clearance between both crucibles, this dispersed around, and the perimeter was polluted with the conventional graphite crucible (example 1 of a comparison). On the other hand, in the case of the graphite crucible of this invention (example 1), in order that gas may fall out from the pore of a pars basilaris ossis occipitalis smoothly, a quartz crucible sinks smoothly, there is [the insertion set was not only made easily, but] almost no blowdown from the upper part of gas, therefore most surrounding contamination was not accepted.
[0019] Next, in order to carry out comparative evaluation of contamination extent in a quartz crucible, the example 1 and the example 1 of a comparison put the pure 5 inch phi silicon wafer on the internal pars basilaris ossis occipitalis of a quartz crucible, the wafers in a crucible were collected after quartz crucible loading, respectively, the number of particle which adhered to the wafer, respectively was measured, and comparative evaluation of extent of contamination was carried out. The result is shown in a table 1. Moreover, when the particle adhering to each wafer was collected and analyzed, carbon was detected at 50% or more of a rate by the wafer adhesion particle in the example 1 of a comparison. On the other hand, in the case of the example 1, most carbon was not detected. The result is shown in a table 1. In addition, the number from which the path made the thing 0.2 micrometers or more the measuring object, and the number of particle before and behind a quartz crucible set increased the measured particle was counted.
[0020]
[A table 1]

	付着パーテイクル数（ΔN）	パーテイクル分析結果
実施例1	10個未満／ウエハ	炭素不検出（検出限界以下）
比較例1	10個以上／ウエハ	炭素検出

[0021] 300mmphi silicon single crystal was pulled up from the quartz crucible which put in melting silicon on the following conditions using the graphite crucible of this invention used in the example 2 and the "example 2 of comparison" example 1, and the conventional graphite crucible of the example 1 of a comparison, respectively (an example 2, example 2 of a comparison). The raising conditions at this time are as follows.
amount of charges (amount of silicon loading to quartz crucible): -- furnace internal pressure: -- Ar gas ambient atmosphere -- raising was carried out 10 times each about each graphite crucible 30 to 100 Torr, and the rate of a defective of the obtained ingot was inspected. About 180kg Raising crystal: <100> Raising rate: 0.3 - 0.8 mm/min Crystal revolution (rotational frequency of quartz crucible): 8 - 15rpm The result is shown in a table 2.
[0022]
[A table 2]

	良品率（%）	備考欄
実施例2	80以上	異物付着による不良品殆ど無し
比較例2	40～60	異物付着で引上初期に不良品頻度大

[0023] The owner rearrangement-ized phenomenon by adhesion of a foreign matter was conventionally seen notably by the ****** case [in the middle of a pull-up] by the elegance crucible (example 2 of a comparison). In almost all cases, carbon was detected when this affix was extracted and analyzed (a foreign matter is presumed to be SiC.). On the other hand, in the case of this invention article (example 2), also when adhesion of a foreign matter was hardly seen but a defective was generated, it was what most depends on other causes.
[0024] Moreover, the wafer was cut down from the tail of the silicon crystal ingot pulled up (three logging from each ingot as samples a, b, c, A, B, and C), and carbon concentration was measured with the infrared-absorption-spectrum measuring method. Consequently, in the case of an elegance crucible, it is 1017 atoms/cm3 conventionally. The carbon of

level was detected. On the other hand, in the case of this invention article, it is 1016 atoms/cm3. It was the following. The result is shown in a table 3.

[0025]

[A table 3]

	サンプル	炭素濃度
実施例2	a	$< 5 \times 10^{15}$ atoms/cm^3
	b	$< 5 \times 10^{15}$ atoms/cm^3
	c	8.1×10^{15} atoms/cm^3
比較例2	A	22×10^{17} atoms/cm^3
	B	6.5×10^{16} atoms/cm^3
	C	1.2×10^{17} atoms/cm^3

[0026]

[Effect of the Invention] Since the silicon wafer crystal-pulling equipment of this invention is what has a specific configuration as the graphite crucible mentioned above, it does so the effectiveness that loading to the graphite crucible of a quartz crucible is easy. Moreover, contamination by the carbon powder of quartz crucible contents is avoided, and it has the advantage of the rate of a defective of the silicon single crystal ingot obtained as compared with the equipment which used the conventional graphite crucible being improved notably, and is especially useful to the diameter silicon single crystal pull-up of macrostomia with a diameter of 300mm or more.

[Translation done.]

* NOTICES *

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] Drawing 1 is cross-section schematic drawing of the graphite crucible for support used with the silicon single crystal raising equipment of this invention.
[Drawing 2] Drawing 2 is cross-section schematic drawing of the graphite crucible for support used with conventional silicon single crystal raising equipment.
[Drawing 3] Drawing 3 is a partly sectional fragmentary schematic illustration of the graphite crucible in which ****** for deflation was prepared further used with the silicon single crystal raising equipment of this invention.
[Drawing 4] It is a schematic diagram for explaining the crystal-pulling equipment used with the Czochrlski method.
[Description of Notations]
1 Body of Crystal-Pulling Equipment
2 Quartz Crucible
3 Graphite Crucible
4 Penetration Opening
5 Striation
6 Heater
7 Heat Insulator
8 Silicon Melt
9 Pull-up Means
10 Seed Crystal
11 Inert Gas
12 Revolution Means

[Translation done.]

* NOTICES *

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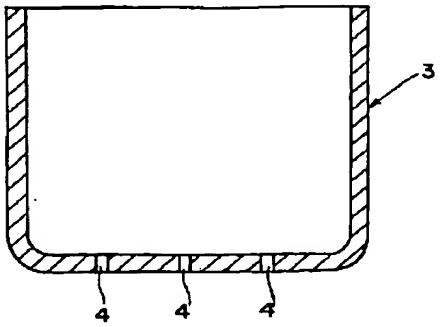
damages caused by the use of this translation.

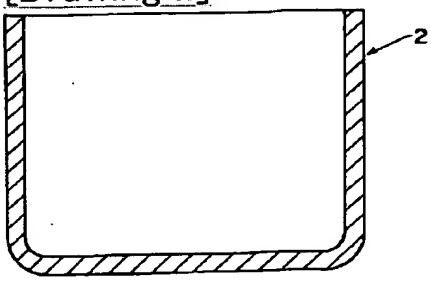
1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DRAWINGS

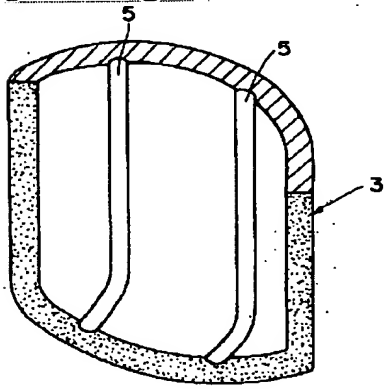
[Drawing 1]



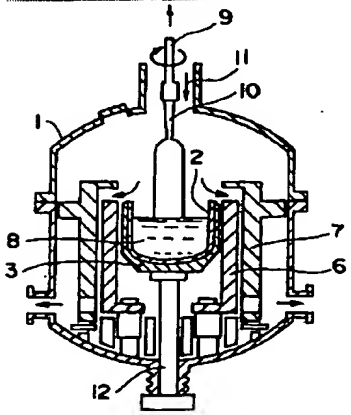
[Drawing 2]



[Drawing 3]



[Drawing 4]



[Translation done.]